

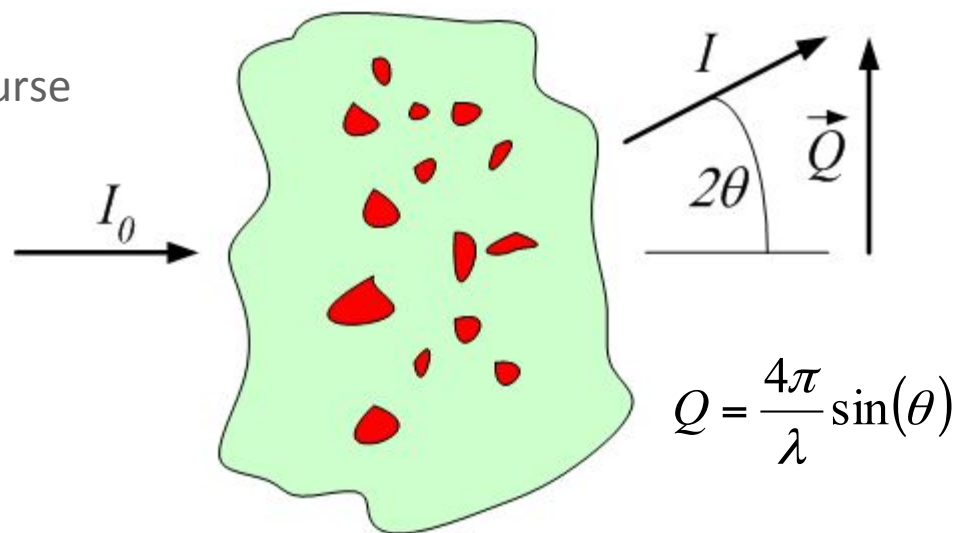
Advanced Photon Source Small-Angle Scattering Instruments

Pete R. Jemian

Small-Angle Scattering Short Course

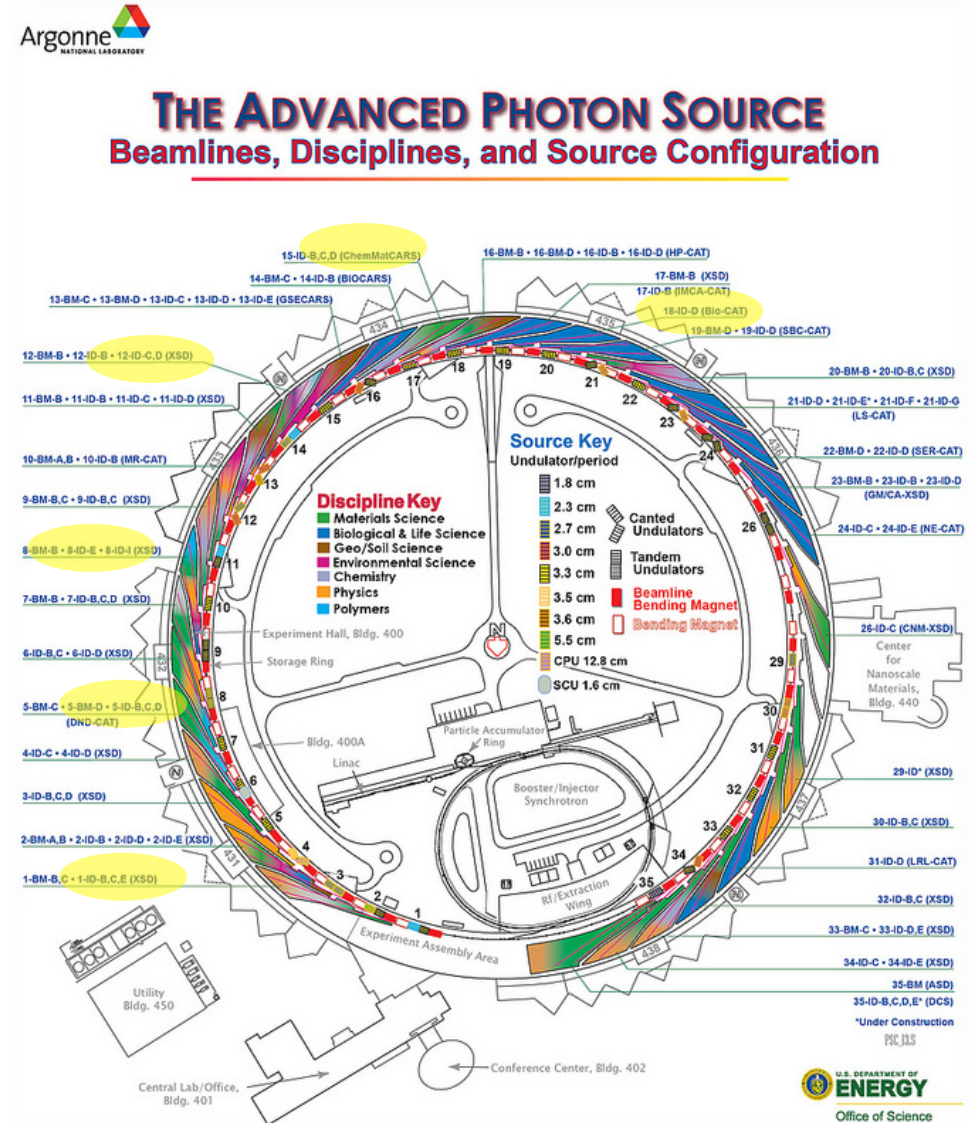
Advanced Photon Source

October 2013



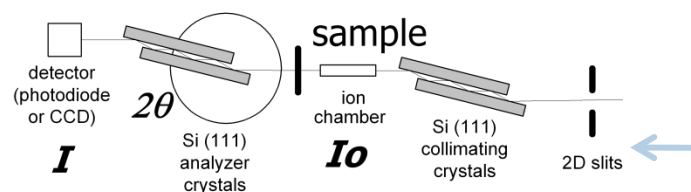
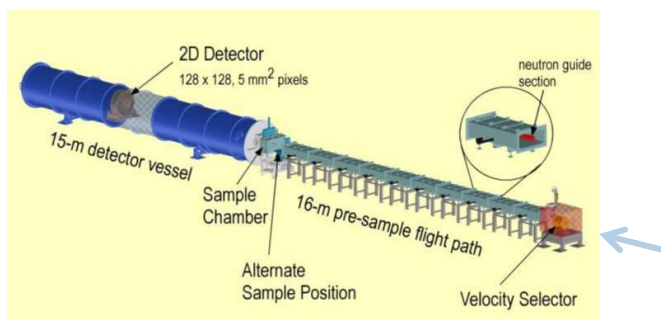
Overview

- Types of SAXS Instruments
- Instrument Selection Criteria
- SAXS Beam Lines at the APS

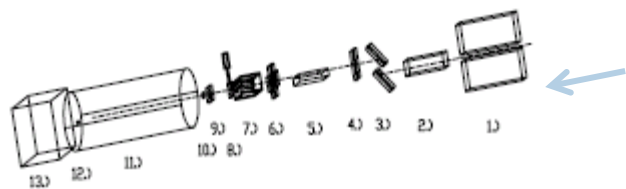


Types of SAXS and SANS Instruments

NIST/NCNR NG-7, pinhole, monochromatic

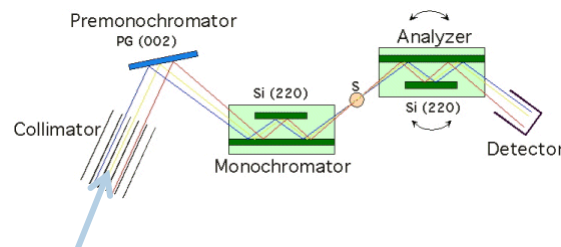


ANL/APS 15ID, Bonse-Hart, monochromatic

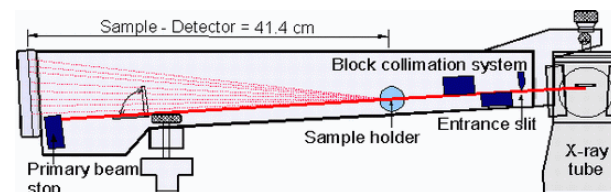


ANL/APS 12ID, pinhole, monochromatic

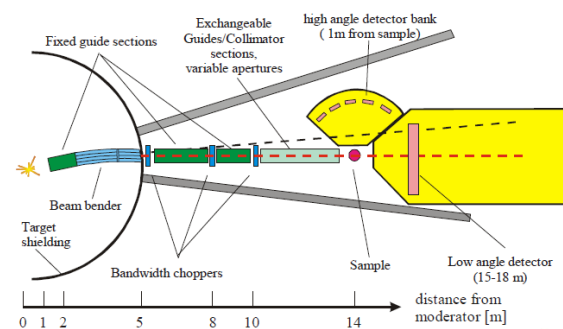
NIST/NCNR, Bonse-Hart, monochromatic



U. Bayreuth, SAXS, Kratky camera

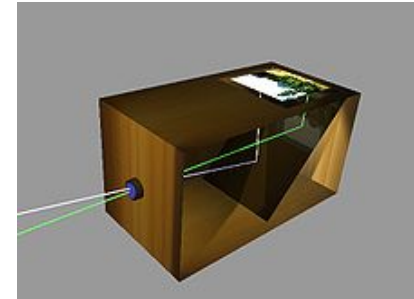


ORNL/SNS EQ-SANS, pinhole, time-of-flight

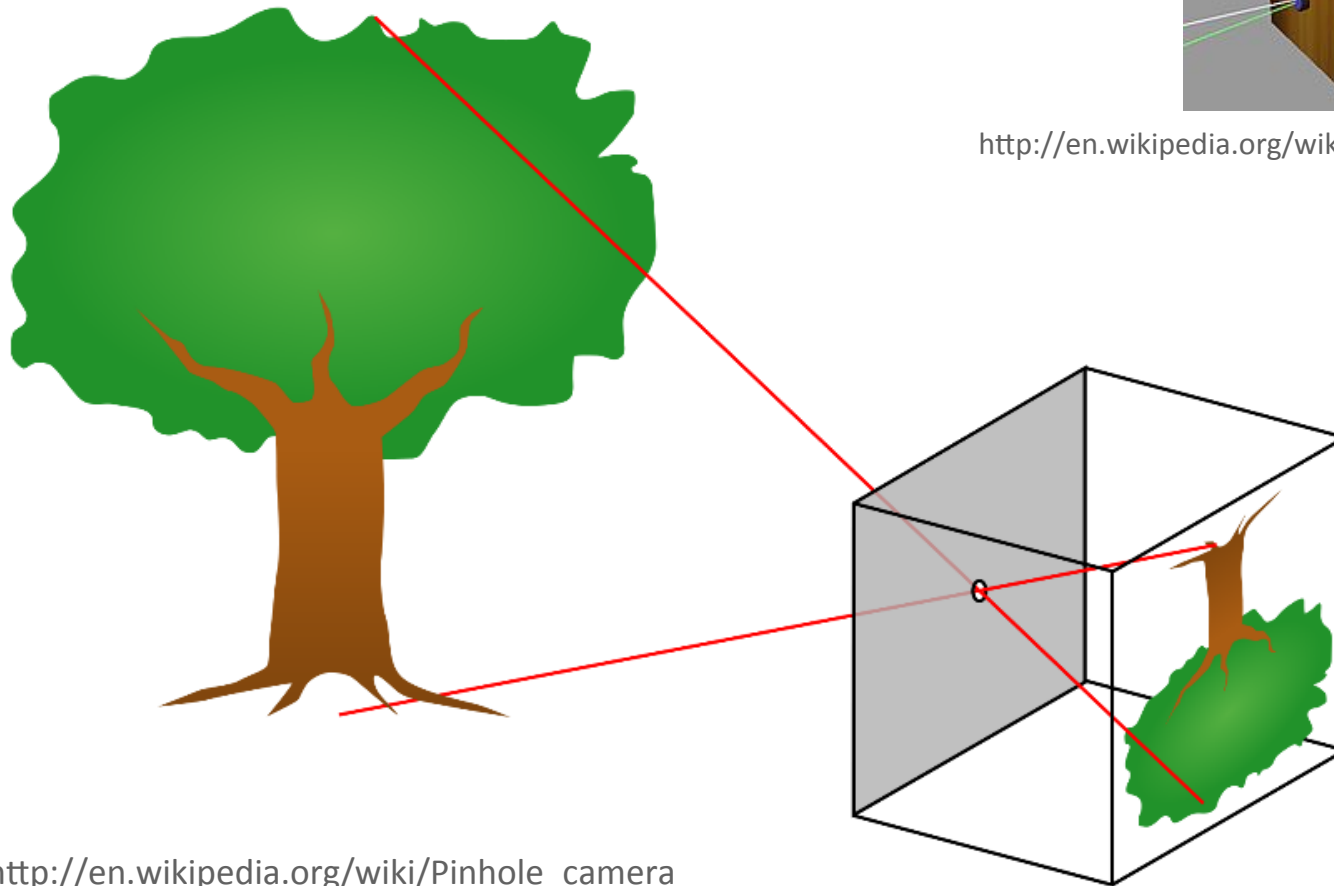


... and there are more (GI-SAXS, XPCS, MIE-SANS, ...)

The pinhole: simplest camera of them all



http://en.wikipedia.org/wiki/Camera_obscura



http://en.wikipedia.org/wiki/Pinhole_camera

How old is the pinhole camera?

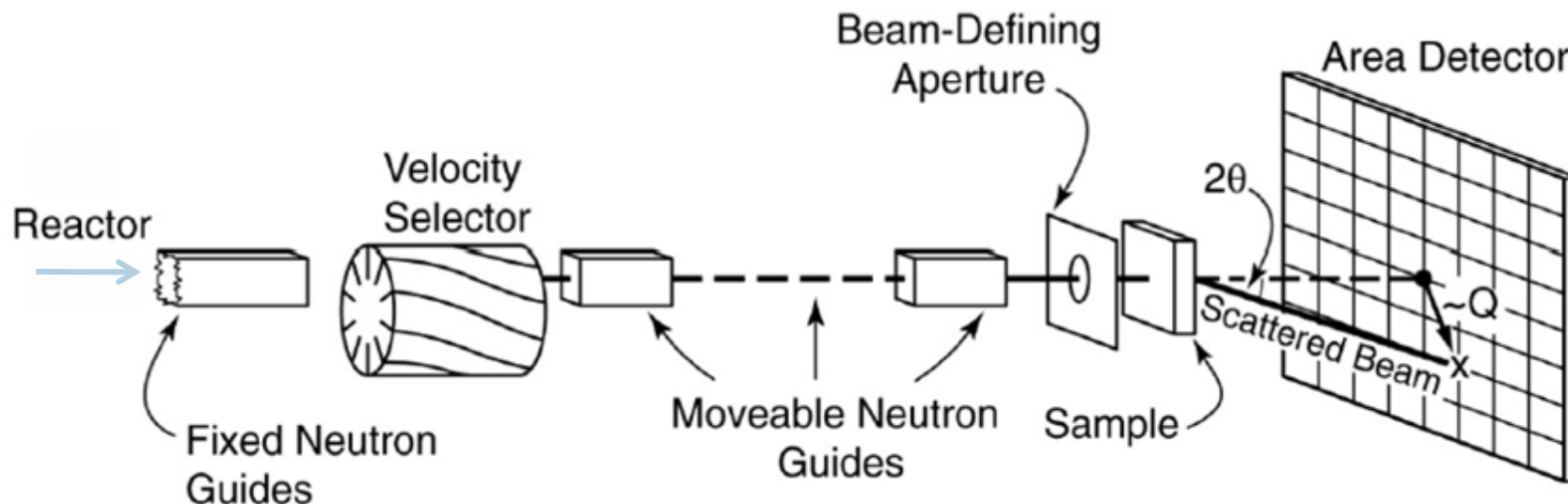
http://en.wikipedia.org/wiki/Pinhole_camera

In the 5th century BC, the [Mohist](#) philosopher [Mo Jing](#) (墨經) in [ancient China](#) mentioned the effect of an inverted image forming through a pinhole.^[2] The image of an inverted [Chinese pagoda](#) is mentioned in [Duan Chengshi](#)'s (d. 863) book [Miscellaneous Morsels from Youyang](#) written during the [Tang Dynasty](#) (618–907).^[3] Along with experimenting with the pinhole camera and the [burning mirror](#) of the ancient Mohists, the [Song Dynasty](#) (960–1279 CE) [Chinese](#) scientist [Shen Kuo](#) (1031–1095) experimented with camera obscura and was the first to establish [geometrical](#) and [quantitative](#) attributes for it.^[3]



An example of a 20 minute exposure taken with a pinhole camera.

Layout of a Reactor SANS pinhole instrument



Recent book

PROBING NANOSCALE STRUCTURES –THE SANS TOOLBOX

http://www.ncnr.nist.gov/staff/hammouda/the_SANS_toolbox.pdf

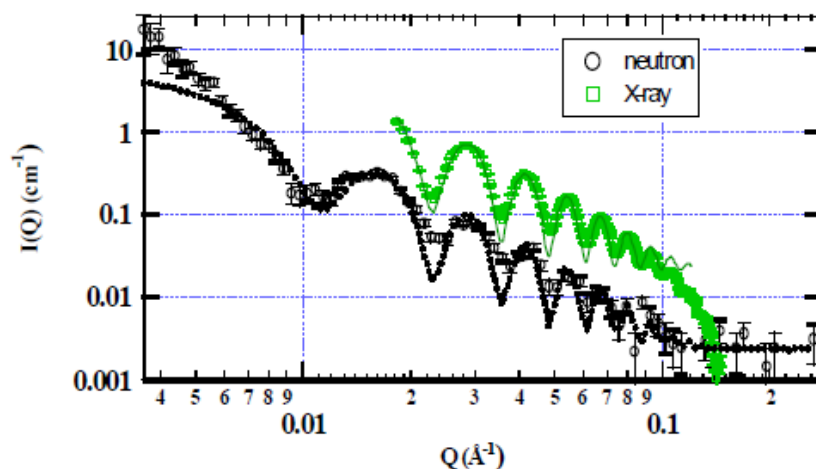
Recent publications:

<http://www.ncnr.nist.gov/programs/sans/publications.html>

Instrument Selection Criteria

- Q range (low Q and Q range)
 - Combined SAXS/WAXS to cover a wide Q range
- Synchrotron vs. Lab source
- Energy Range, Access to different energies as needed
- Flux at the sample
- Detectors
- Ancillary Equipment
 - Flow cell, FPLC, mechanical test rig, stop flow and continuous flow mixers, heater, cooler, chiller, flame pyrolysis, ..., exhaust gas handling
 - Automated sample changer, versatile sample environment
- Specialty of Beam line Scientists
- User Support
 - Lab facilities, software suite for data reduction and analysis, specialized computing
- Data Reduction, Analysis
- Radiation Damage mitigation
- Contrast Variation Studies
 - SANS vs. ASAXS or both for Multi-component systems

Synchrotron SAXS offer Better ΔQ resolution



Peptide nanotubes:

Kun Lu, Jaby Jacob, P. Thiyagarajan,
Vincent P. Conticello, David
Lynn, JACS (2003), 125, 6391-
6393.

$$\left(\frac{\Delta Q}{Q}\right)^2 = \left(\frac{\Delta \theta}{\theta}\right)^2 + \left(\frac{\Delta \lambda}{\lambda}\right)^2$$

*hollow cylinder
form factor:*

$$P(Q) = \frac{1}{\left[1 - \left(\frac{R_2}{R_1}\right)^2\right]^2} \left[\frac{2J_1(QR_1(1-x^2)^{0.5})}{QR_1(1-x^2)^{0.5}} - \left(\frac{R_2}{R_1}\right)^2 \frac{2J_1(QR_2(1-x^2)^{0.5})}{QR_2(1-x^2)^{0.5}} \right]^2 \left(\frac{\sin(QH_x/2)}{QH_x/2} \right)^2 dx$$

SANS

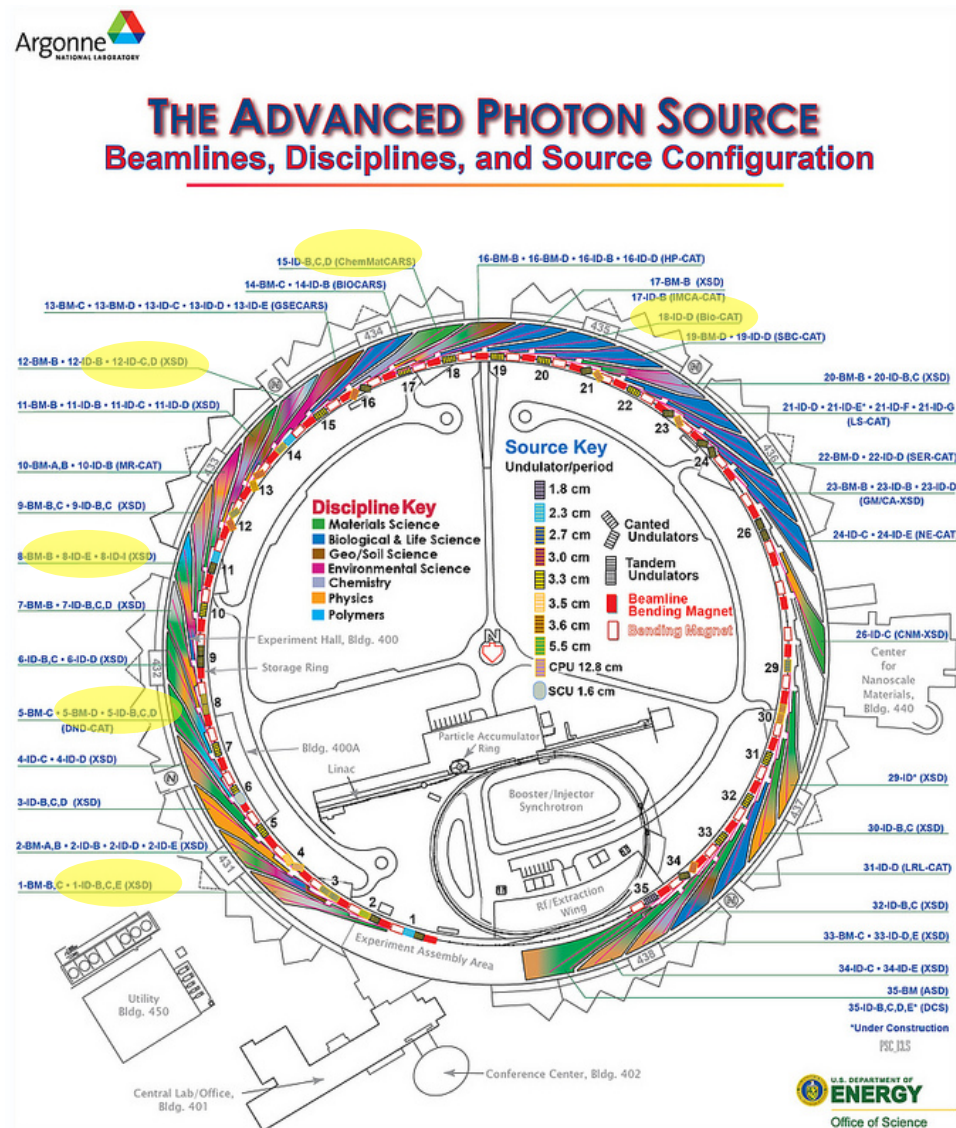
outer $R_1 = 259.37 \pm 1.33 \text{ \AA}$
inner $R_2 = 216.03 \pm 0.71 \text{ \AA}$
wall thickness = 43.3 \AA

SAXS

outer $R_1 = 266.01 \pm 0.01 \text{ \AA}$
inner $R_2 = 224.64 \pm 0.03 \text{ \AA}$
wall thickness = 41.4 \AA

SAXS Beam Lines at the APS

- **1ID High-Energy SAXS/WAXS**,
Jon Almer
- **5ID SAXS-WAXS**,
Stephen Weigand
- **8-ID-E GISAXS**,
Jin Wang, Joe Strzalka, Zhang Jiang
- **8-ID-I XPCS**,
Alec Sandy, Suresh Narayanan
- **12-BM SAXS**,
Sungsik Lee, Randy Winans
- **12-ID-B GISAXS**,
Byeongdu Lee, Xiaobing Zuo
- **12-ID-C SAXS**,
Sönke Seifert, Randy Winans
- **15-ID USAXS**,
Jan Ilavsky
- **18-ID SAXS**,
Tom Irving, Rita Graceffa



APS Small Angle X-ray Scattering (SAXS) Beamlines

(http://small-angle.aps.anl.gov/aps_beam_lines.html)

| beam line | experiments | Energy (keV) | Q (1/Å) (*) | Operating group | Contact |
|-----------|---|---------------------------------------|-----------------------|-----------------|--|
| 1-ID | High-Energy SAXS Simultaneous SAXS/WAXS | 50 - 100 | 0.005 - 0.4 1 - 10 | XSD-MPE | Jon Almer, 630-252-1049 <almer@aps.anl.gov> |
| 5-BM | High-Energy SAXS | 5 - 65 8 - 20 with focusing | 0.008 - 20 | DND | Qing Ma, 630-252-0229 <q-ma@northwestern.edu> |
| 5-ID | Simultaneous SAXS/WAXS Windowless sample changer Polymer stress-strain (Instron) DSC (Linkam) solution scattering | 5 - 19 | 0.001 - 5 | DND | Steven Weigand, 630-252-0623 <weigandsj@northwestern.edu> |
| 8-ID-E | GIXS and reflectivity | 7.35 (usually) or 12 | 0.005 - 2.5 | XSD-TRR | Joseph Strzalka, 630-252-0283 <strzalka@aps.anl.gov> Zhang Jiang, 630-252-3118 <zjiang@aps.anl.gov> |
| 8-ID-I | XPCS, SAXS, time-resolved SAXS (collaboration only) | 6 - 12.5 (generally fixed at 7.35) | 0.002 - 0.15 | XOR-TRR | Alec Sandy, 630-252-0281 <asandy@anl.gov> Suresh Narayanan, 630-252-0287 <sureshn@aps.anl.gov> |

(*) *Approximate* Q ranges are given, depending on conditions; contact the beam line for more information

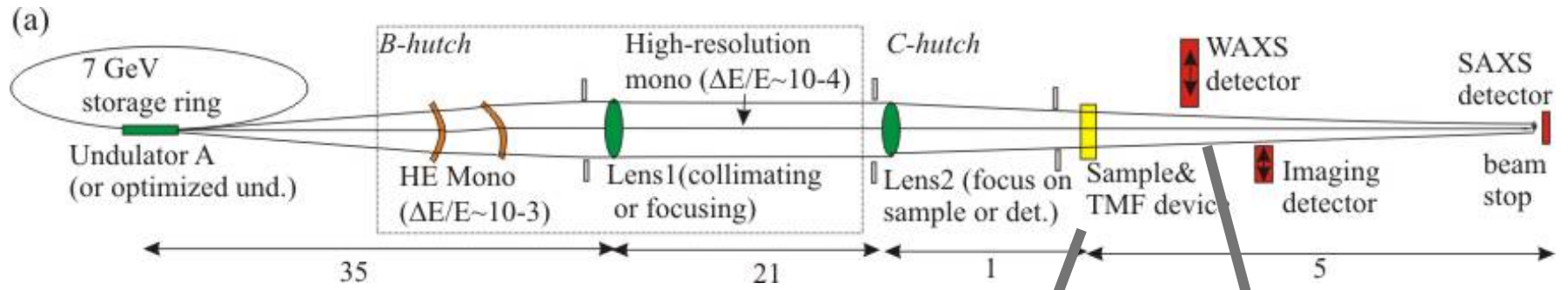
APS Small Angle X-ray Scattering (SAXS) Beamlines

(http://small-angle.aps.anl.gov/aps_beam_lines.html)

| beam line | experiments | Energy (keV) | Q (1/Å) (*) | Operating group | Contact |
|-----------|---|--------------|--------------|------------------|---|
| 12-BM | SAXS, Solution Scattering | 5 - 22 | 0.001 - 1 | XSD-CMS | Sungsik Lee, 630-252-7491 <sungsiklee@anl.gov> |
| 12-ID-B | GISAXS, Solution SAXS, SAXS/WAXS, bio SAXS | fixed at 12 | .005 - 3 | XSD-CMS | Byeongdu Lee, 630-252- <blee@aps.anl.gov> Xiaobing Zuo, 630-252-1553 <zuox@aps.anl.gov> |
| 12-ID-C | SAXS/WAXS, time-resolved SAXS, ASAXS | 4.8 - 25 | 0.0012 - 2.8 | XSD-CMS | Sönke Seifert, 630-252-0391 <seifert@anl.gov> |
| 15-ID-D | USAXS (1-D or 2-D), USAXS Imaging, Anomalous USAXS | 7 - 18 | 0.0001 - 1 | XSD, ChemMatCARS | Jan Ilavsky, 630-252-0866 <ilavsky@anl.gov> http://usaxs.xor.aps.anl.gov |
| 18-ID | SAXS/WAXS, static and time-resolved, biological samples, solutions, 2-D focused beams | 3.5 - 40 | 0.002 - 3.0 | BioCAT | Tom Irving, 630-252-0524 irving@biocat1.iit.edu Rita Graceffa, 630-252-0549 graceffa@bio.aps.anl.gov |

(*) Approximate Q ranges are given, depending on conditions; contact the beam line for more information

1-ID: HE-SAXS/WAXS

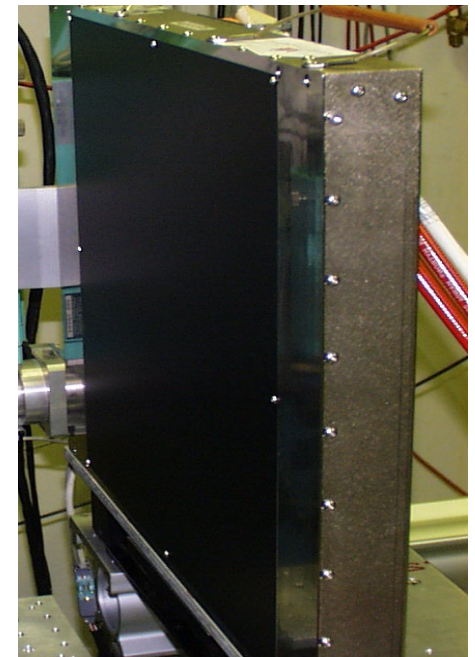
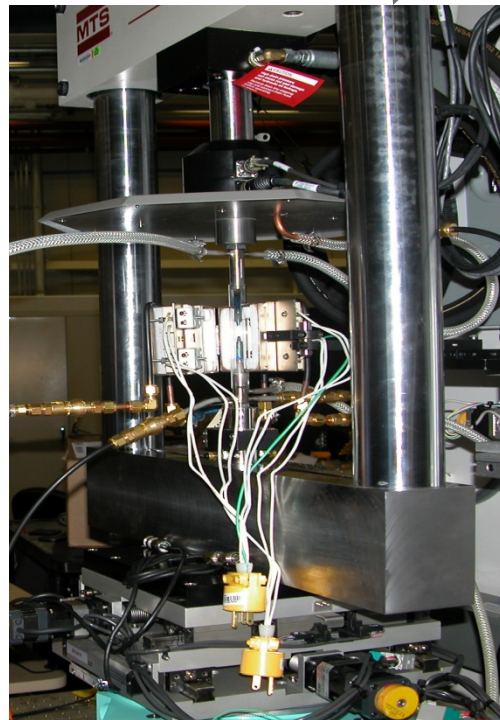


Multiple modes

- WAXS (down to $d \sim 1$ Å)
- SAXS (up to $d \sim 5000$ Å)
- Imaging / radiography
- Fluorescence

Transverse beam size down to $\sim 1 \times 10 \mu\text{m}^2$ using lenses

Diffraction tomography under development

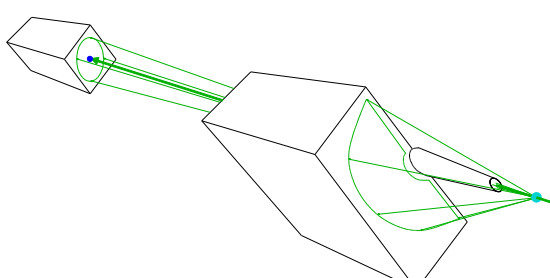
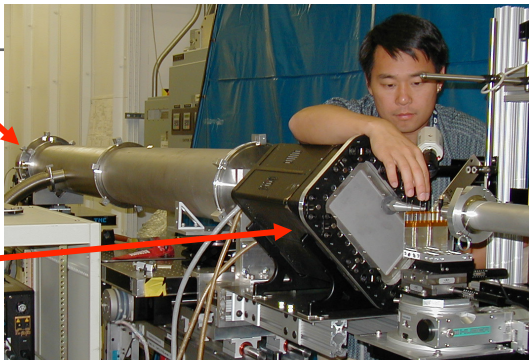


SAXS at DuPont-Northwestern-Dow CAT's 5ID-D station

5ID-D SAXS Parameters

| | |
|--|---|
| APS Undulator A insertion device | |
| Si(111) monochromator | 7 keV to 18 keV (1.8 Å to 0.7 Å) |
| 1:1 horizontal bent mirror focusing | Rh, Glass, or Pt |
| Standard slit-based pinhole geometry | Beam size → 50 μm × 50 μm to 1 mm × 2.5 mm |
| Wide range of camera lengths available | 136 mm to 10,000 mm |
| Momentum transfer over three orders | 0.001 Å ⁻¹ to 5 Å ⁻¹ (6200 Å to 1.26 Å) |

Detectors

| | | |
|--|---|--|
| Rayonix Mar165 phosphor fiber-optic coupled CCD |  |  |
| Custom Roper SAXS/WAXS phosphor fiber-optic coupled CCD system | | |

SAXS detector WAXS detector

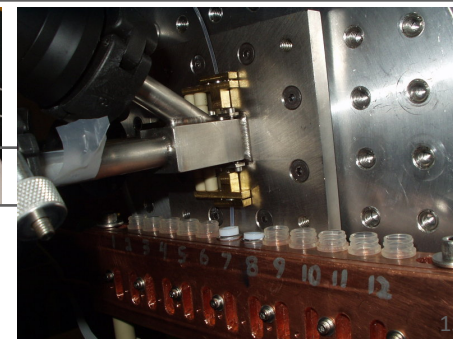
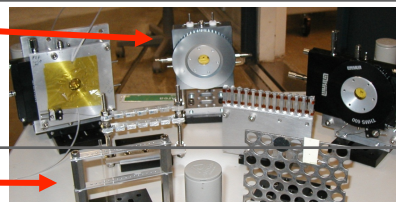
Sample Environments

Large translation stages and open sample area for custom environments

Temperature stages from Linkam:
DSC, capillary, capillary flow-cell
2 cm diameter area, -196 °C to 600 °C

Numerous static multi-sample stages

Vacuum spanning capillary flow-cell
water cooled multi-sample changer
solvent compatible (all Teflon® and quartz)



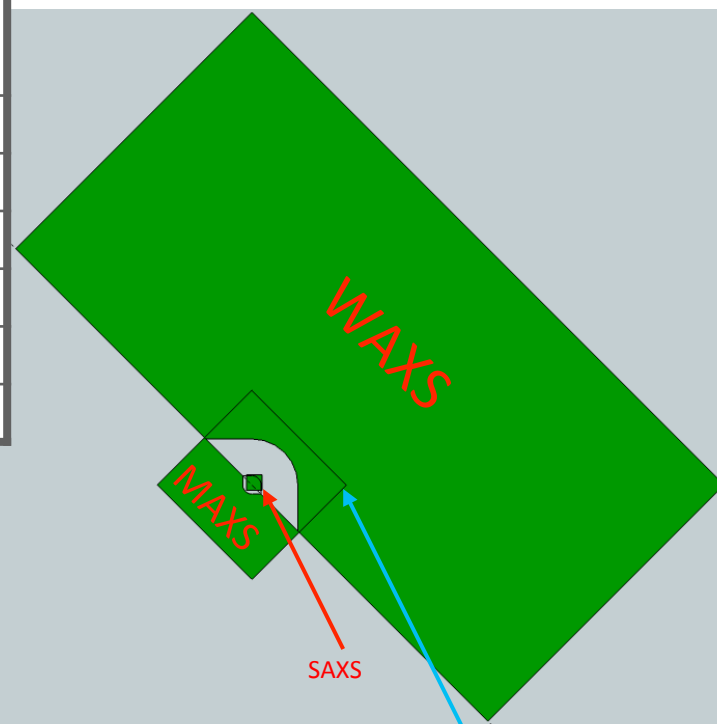
2013-10-2



SAXS at DuPont-Northwestern-Dow CAT's 5ID-D station

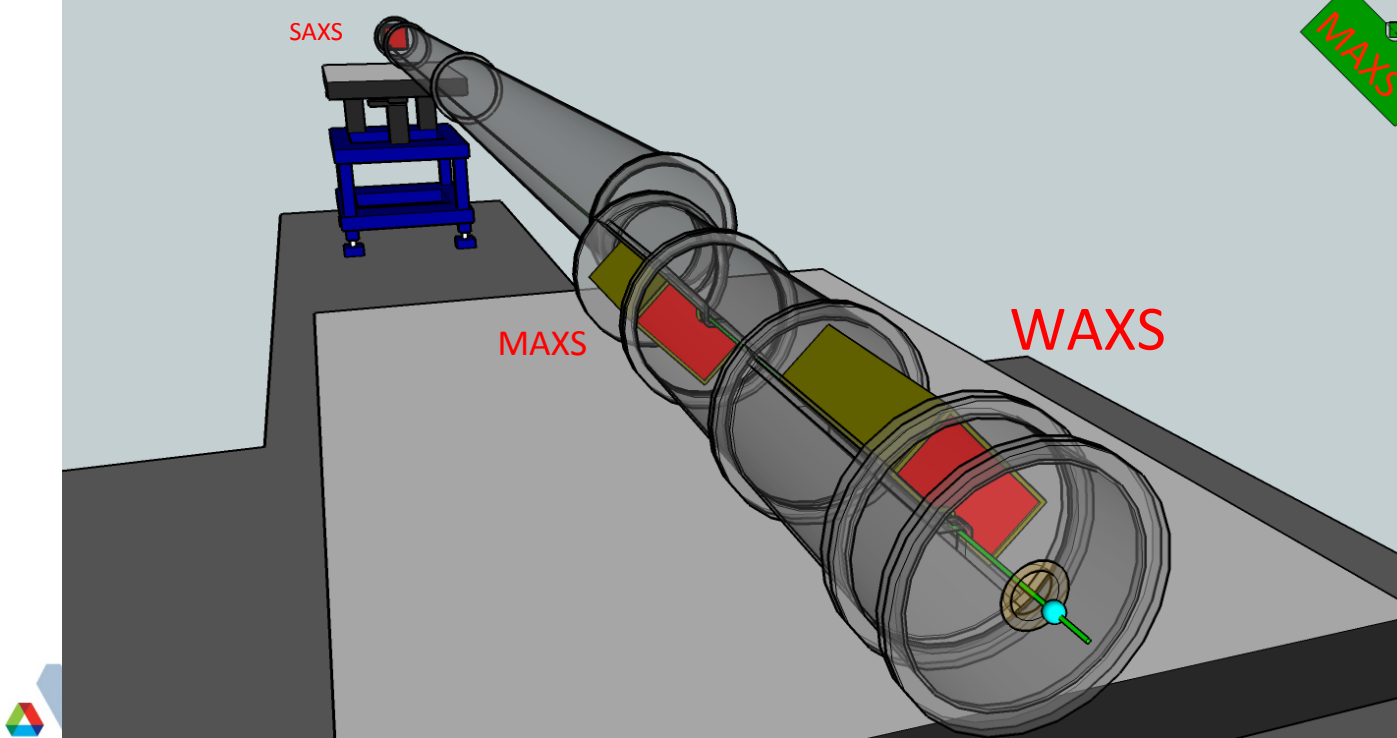
Coming Soon High speed SAXS/MAXS/WAXS

| |
|--|
| 3 simultaneous CCD detectors from Rayonix (LX-HS at 0.2m & 1m; SX-HS at 8.5m) |
| Overlapping momentum transfer ranges over three orders |
| Broad azimuth ranges on 2-D detectors |
| 85 μm pixels at 2×2 binning |
| SAXS $\rightarrow 170 \text{ mm} \times 170 \text{ mm}$ |
| MAXS & WAXS $\rightarrow 85 \text{ mm} \times 170 \text{ mm}$ |
| 10 fps at 2×2 binning; 100 fps at 8×8 binning; 1 ms dead time |

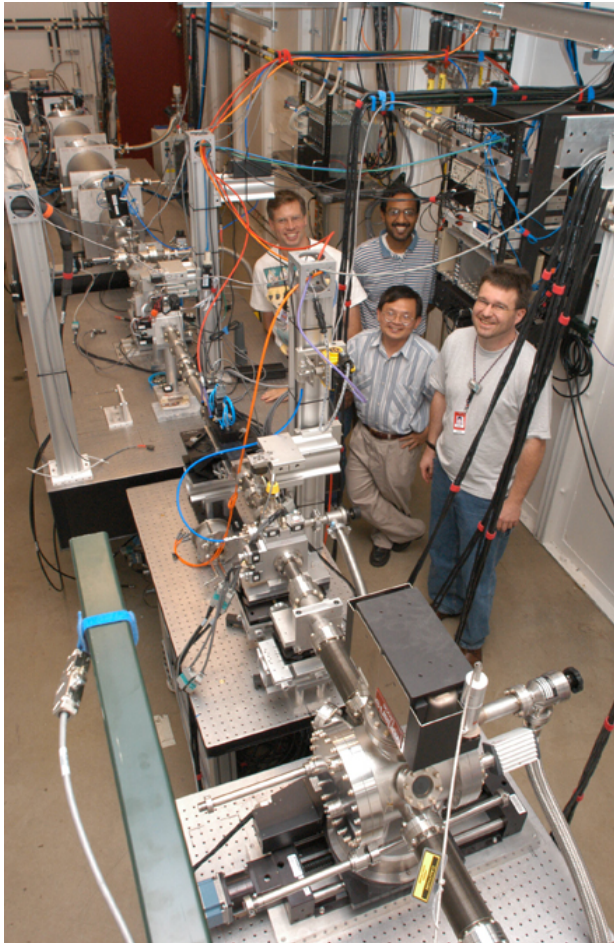


Outline of MAXS
symmetry overlap with
WAXS and SAXS

Beam path is evacuated,
but detectors are in air with
Kapton® windows for each



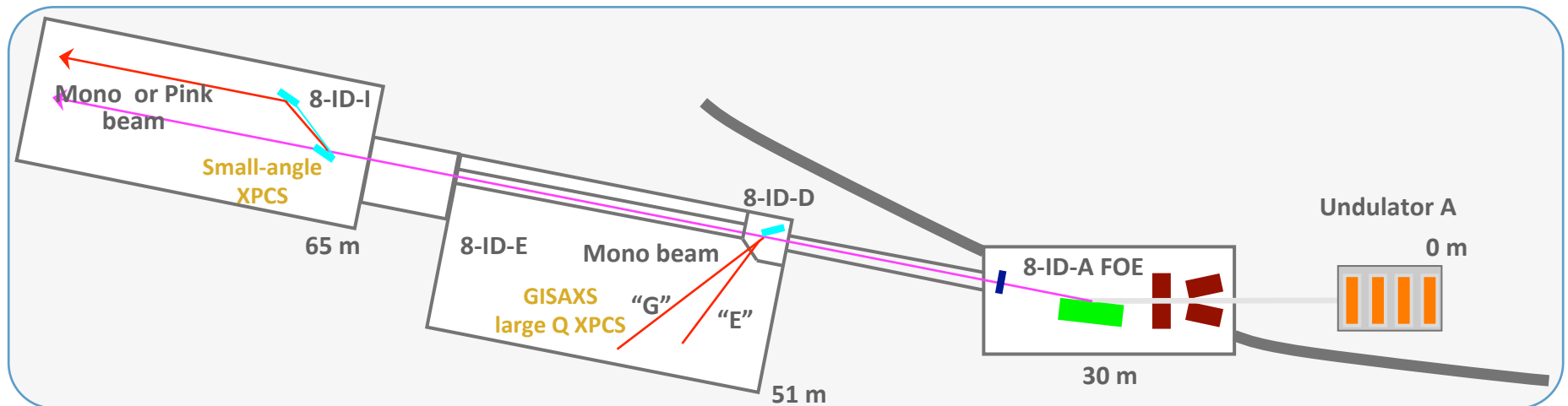
8-ID has XPCS and GIXS



- Part of the APS Time Resolved Research (TRR) Group managed by Jin Wang
- Fully operational with 80% General User (GU) time each cycle
- Staffed with 4 beam line scientists with complementary expertise in the beam line's scientific theme areas
 - Suresh Narayanan
 - Alec Sandy
 - Joseph Strzalka
 - Zhang Jiang

8-ID layout

- Undulator beam line supporting 2 scientific theme areas:
 - Grazing-Incidence X-ray Scattering (GISAXS)
 - \approx 80% General User (GU) time
 - Dedicated in-vacuum set-up
 - X-ray photon correlation spectroscopy (XPCS)
 - \approx 80% General User (GU) time
 - Only other similar facility worldwide is at the ESRF
- Most 8-ID user groups from physics, chemistry, chemical engineering, polymer and materials science and engineering disciplines

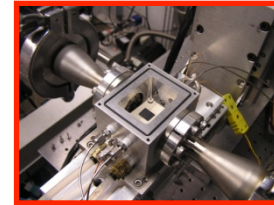


12-ID: *Two Beamlines with Canted Undulators* materials science, chemistry

Slide(s) courtesy of Randy Winans

■ 12-ID-B (7.4 – 13.9 keV, currently fixed at 12 keV)

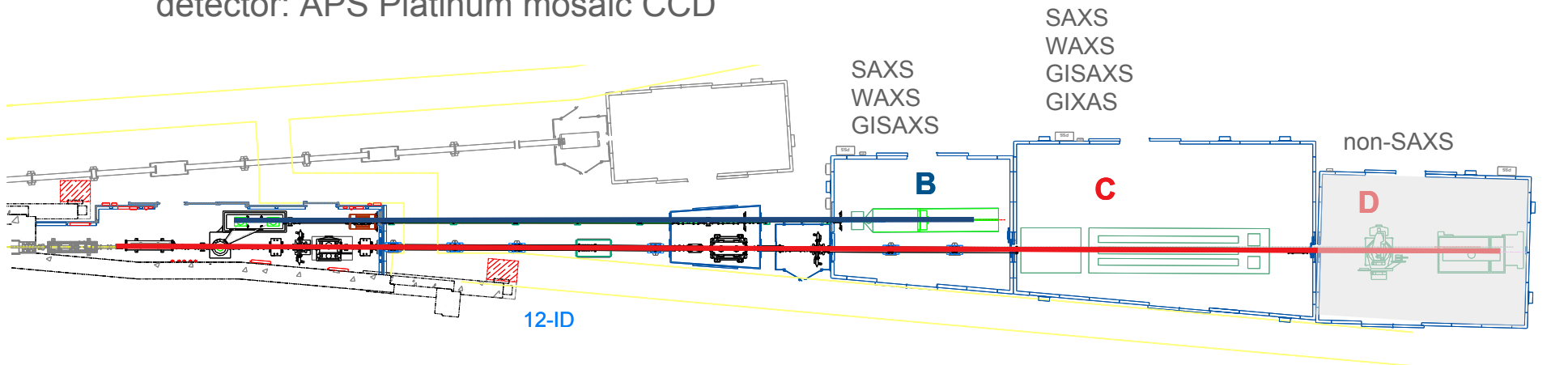
SAXS/WAXS/GISAXS - rapid adjustable Q [12-ID-B]
detectors: Pilatus 2M and wide angle (300K)



GISAXS Cell

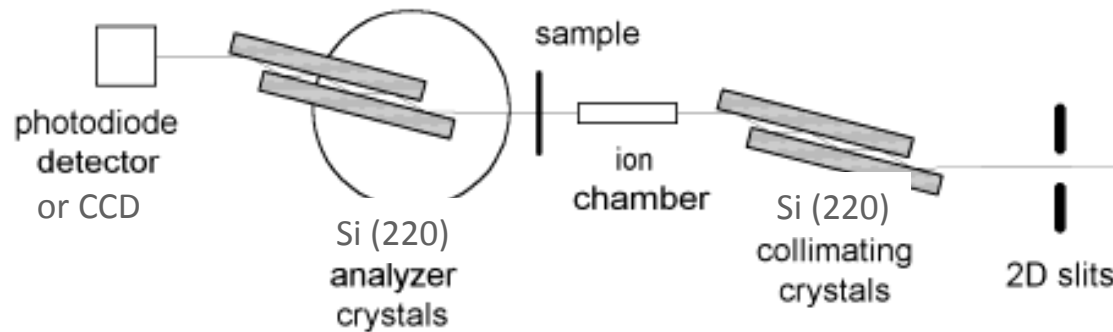
■ 12-ID-C (4.5 – 36 keV, pink beam)

SAXS/WAXS/GISAXS/GIXAS - in situ, time resolved [12-ID-C]
Surface Scattering – MOCVD, surface diffraction [12-ID-D]
detector: APS Platinum mosaic CCD

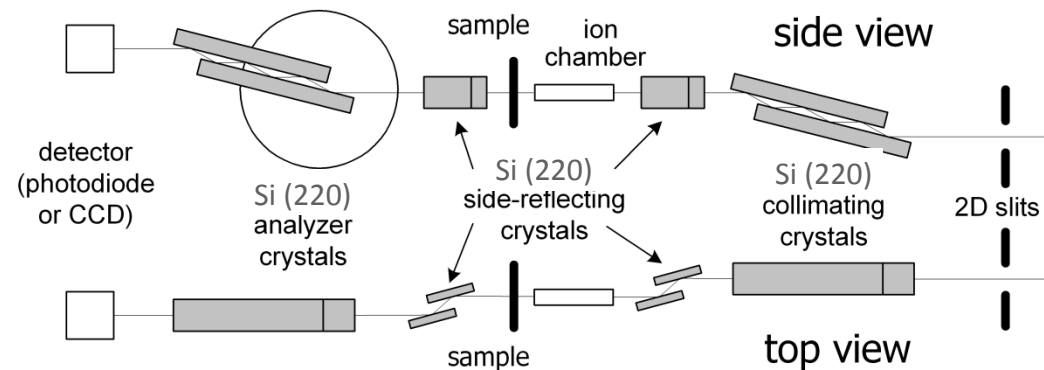


15-ID-D USAXS

materials science, larger structures, USAXS imaging



1-D collimated Bonse-Hart Camera (slit smeared)

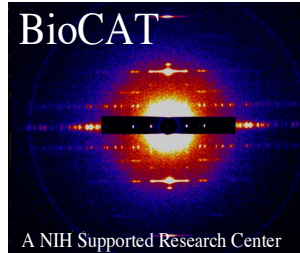


2-D collimated Bonse-Hart Camera

15-ID-D USAXS parameters

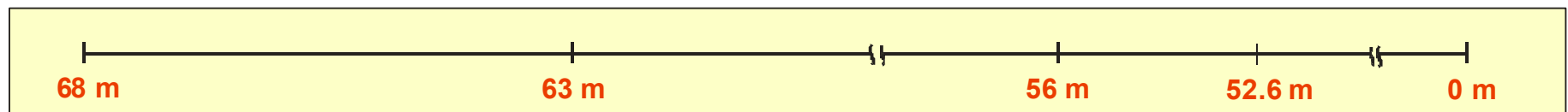
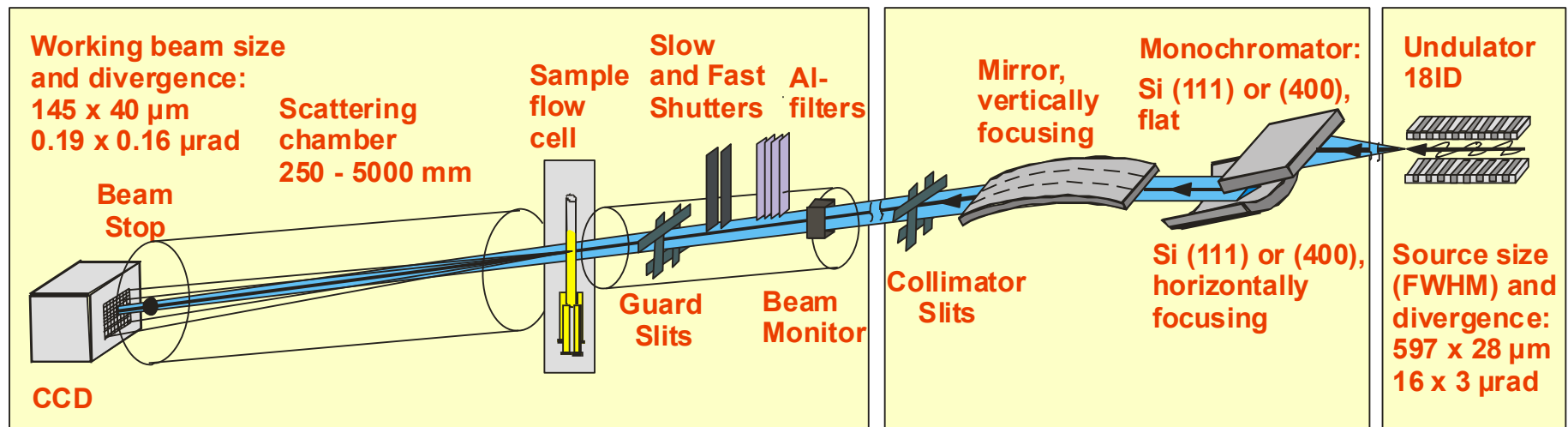
- General purpose USAXS instrument using Bonse-Hart design
- Used to examine structures in the few micrometer scale
- Intensity and Q range:
 - ca. 10^{13} ph/s (monochromatic) incident on sample
 - Up to 9 decades of intensity range
 - 0.0001 \AA^{-1} to 1 \AA^{-1} Q range (0.5 nm ----> >1 micron)
 - Both 1-D (slit smeared) and 2-D collimated (“2D-USAXS”) geometries available
 - 10 min/scan (shortest scans down to 3 minutes)
 - Flexible beam size (1 x 2 mm ----> 0.02 x 0.2 mm)

18-ID SAXS biology, solutions



Slide(s) courtesy of Tom Irving

- A NIH-supported research center for the study of partially ordered and disordered biological materials. Operated by the Illinois Institute of Technology
- Comprises an undulator based beamline, (18-ID) associated laboratory and computational facilities.
- Available to all scientists on basis of peer-reviewed beam time proposals



18-ID BioCAT parameters

- Total X-ray flux $1\text{-}2.5 \times 10^{13}$ photons/s
- Focal spot size ranges from $< 50 \mu\text{m}$ vertical and $< 160 \mu\text{m}$ horizontal to $\sim 3 \times 1.5$ mm
- Wide energy range (4-39 keV)
- Rapid 1 keV energy scans in < 15 seconds
- First order resolution $> 1500 \text{ \AA}$ (1/d)
- Order to order resolution $> 10000 \text{ \AA}$ (1/d)
- High sensitivity (\sim photon counting), high spatial resolution (~ 60 micron psf, 39 micron pixels) CCD detectors
- Pilatus 100k detector for time resolved studies

Thank you for your attention!



Acknowledgments

- APS Instrument Scientists
 - Jon Almer, Rita Graceffa, Liang Guo, Jan Ilavsky, Tom Irving, Zhang Jiang, Qing Ma, Byeongdu Lee, Sungsik Lee, Suresh Narayanan, Alec Sandy, Sönke Seifert, Joseph Strzalka, Jin Wang, Steven Weigand, Randy Winans, Xiaoqing Zuo
- Thiya P. Thiyagarajan, US Dept. of Energy, Boualem Hammouda, NIST, Jinkui Zhao, ORNL

